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Stability of Bulk $Ba_2YCu_3O_{7-x}$ in a Variety of Environments

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STABILITY OF BULK $Ba_2YCu_3O_{7-x}$ IN A VARIETY OF ENVIRONMENTS

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SUMMARY

Small bars (1 by 1 by 14 mm) of ceramic $Ba_2YCu_3O_{7-x}$ were fabricated and subjected to environments similar to those that might be encountered during some NASA missions. These conditions include ambient conditions, high humidity (100 percent humidity up to 50 °C), vacuum (10^{-4} Pa), and high fluences of electrons (9.7×10^{14} e^-/cm^2) and protons (8.7×10^{14} p^+/cm^2). The normal state resistivity or critical current density (J_c) were monitored during these tests to assess the stability of the material under these conditions.

When normal state resistivity is used as a criterion, the ambient stability of these samples was relatively good, exhibiting only a 2 percent degradation over a 3 month period. The humidity stability was shown to be very poor, and to be a steep function of temperature. Samples stored at 50 °C for 40 min increased in normal state resistivity by four orders of magnitude. Kinetic analysis indicates that the degradation reaction is second order with water vapor concentration. It is suspected that humidity degradation also accounts for the ambient instability. The samples were stable to vacuum over a period of at least 3 months.

Degradation of J_c (defined as the current density that produced a potential greater than 1 μV/mm) in an 1 MeV electron fluence of 9.7×10^{14} e^-/cm^2 was determined to be no more than about 2 percent. Degradation of J_c in a 8.7×10^{14} p^+/cm^2 of 42 MeV protons was found to be grain size dependent. Samples with smaller grain size and initial J_c (77 K) of about 240 A/cm^2 showed no degradation, while that with larger grain size and an initial J_c (77 K) of about 30 A/cm^2 degraded to 37 percent of its original value.

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INTRODUCTION

It is commonly believed that space technology will be one of the first arenas where the perovskite-type high temperature superconductors will see their applications. This is because component costs are not as critical as performance, since their costs are likely to be a small fraction of the mission, while mission success may hinge on their performance.

Materials which are used in spacecraft must be able to withstand a wide variety of conditions. These include typical earth environmental factors, such as atmospheric oxygen and water, which any material must either be subjected to or protected from during manufacture and assembly. Launch conditions add to these factors vibrational stresses and high g-force structural loads. If the payload is in a low earth orbit, there is moderately high vacuum (10^{-5} Pa) and the residual atmosphere is dominated by a very corrosive gas, atomic oxygen. At high earth orbits and deep space there is high vacuum (10^{-8} Pa), and radiation fluxes from the solar wind. The radiation fluxes are particularly high in the radiation belts which surround the earth and other planets. If the spacecraft is a planetary probe, it must be able to withstand temperatures, pressures and atmospheric constituents of the target planet. These range from the hot oxidizing environment of Venus, to the cold reducing environments of Jupiter and the other gas giants.

If the intent is to utilize high temperature superconductors on spacecraft, it must first be determined how vulnerable they are to this variety of conditions. If the materials are vulnerable, additional measures which are required to protect the material may be determining factors as to whether an application is feasible. This study is an initial attempt to assess the vulnerability of one of the high temperature superconducting materials, $Ba_2YCu_3O_{7-x}$, to some of these conditions.

There were several limitations to this study which should be recognized from the outset. The first is that the materials investigated were experimental. There are relatively large batch to batch variations and probably some minor phases are present (below the limit of detection using x-ray diffraction). When a standard material becomes available it will no doubt be of higher quality. Since this study used ceramic materials, and not single crystals, some of the observed effects may be artifacts of grain boundaries which will change as the material quality goes up. Nevertheless, it seems reasonable to begin to screen these materials at this early stage to point out possible trouble areas, and areas in which even these crude materials perform well.

METHODS AND MATERIALS

Powders of $Ba_2YCu_3O_{7-x}$ were synthesized with BaO_2, Y_2O_3, and CuO using standard techniques (ref. 1). For comparison, powders were also provided by Ferro Corp. The powders were pressed into bars measuring 1 by 1 by 14 mm at room temperature using a pressure of 200 to 350 MPa. The bars were sintered at 900 °C for 16 hr, and then annealed at 450 °C in an oxygen flow for 8 hr. Samples were stored in a desiccator until used.

Two types of contacts were used, depending on the sample properties to be measured. For the samples which only had T_c and normal state resistivity

measured, silver paint was used to paint contacts on the bars after they were annealed. For those which had J_c measured, silver epoxy contacts were applied after sintering, and were then annealed on.

The resistivity of the samples was measured using conventional four point techniques with a Keithley Model 220 constant current source and a Keithley Model 181 nanovoltmeter. The voltage was measured at both polarities to negate electrochemical effects.

Even though normal state resistivity itself is not a technologically useful parameter with these materials, we have found that it is indicative of the superconducting properties. Samples with deteriorating normal state properties will also have deteriorating superconducting properties. Moreover, normal state resistivity can be measured in-situ, and does not subject the sample to temperature cycling, which we suspect may degrade the sample. We believe the normal state resistivity to be a fairly sensitive indicator of the condition of the sample, since it is measured from the surface where the effects of degradation would most likely be seen first. However, since these samples have much smaller surface to volume ratios than would be encountered in thin films, thin film degradation would no doubt occur much faster.

The transport critical current density (J_c) was measured at 77 K with the sample submerged in liquid nitrogen using a Keithley Model 228 constant current source, and a Keithley Model 181 nanovoltmeter under computer control. Current-voltage (I-V) curves were generated by stepping the current in increments of 0.01 A from the normal state of one polarity through the superconducting transition and back to the normal state at the other polarity. Sample holders were designed such that the sample could be subjected to the environmental condition and then measured repeatedly without having to remount the sample.

The authors believe that transport J_c at 77 K is the critical parameter to be monitored. Since devices will probably be used at this temperature and since J_c is the limiting parameter for these applications, this measurement is the most stringent. J_c was defined in these experiments as the current density that produced a potential greater than 1 μV/mm of length. J_c was also found to be much more sensitive to the effects of degradation than critical temperature (T_c). This is because a sample will show zero resistance at T_c if there is any connecting conduction path between the voltage probes, but J_c is dependent upon the amount of material which is superconducting. Transport J_c is not only the parameter that is required for engineering specifications, but it gives insights into the grain boundary interactions. Grain boundary interactions dominate the superconducting properties of ceramic materials.

After it was confirmed that the samples were superconducting, and had T_c in excess of 90 K, they were mounted on four point resistivity sample boards (fig. 1). The samples were then left out in the ambient air and there normal state resistivity was measured at increasing time intervals.

Additional samples were mounted on sample boards which were attached to vacuum feed-throughs. The feed-throughs were mounted to a vacuum line and evacuated with a turbomolecular pump (TMP) to a pressure of about 10^{-5} Pa. This is approximately the vacuum of low earth orbit. The four point resistivity of the samples could be measured without removing them from vacuum. After

17 days there was a failure of the TMP. So data beyond this time represents exposure to a roughing vacuum of about 10^{-3} Pa.

Other samples were mounted on sample boards and placed in humidity chambers (fig. 2). These consist of two beakers, one with the other. The smaller beaker was partially filled with water, and the sample sat on top of it. This was placed with a larger beaker with the electrical leads trailing out and the large beaker was sealed with Parafilm. This assembly was in turn placed within an incubator set at the test temperature. The chambers were allowed to equilibrate overnight at the test temperature before the samples were added. A chart recorder was connected to the nanovoltmeter because of the speed of the reaction.

Electron irradiations were carried out at the Westinghouse Research Center in Pittsburgh with the Van de Graaff generator. Electrons of 1 MeV energy were used and the flux was measured using standard Faraday cup techniques. Control samples were exposed to the same environment as the test samples, less the radiation. Samples were irradiated to a total fluence of 9.7×10^{14} e^-/cm^2 in three irradiations over a one month period. J_c's of the samples were measured after fluences of 7×10^{13} and 2.7×10^{14} e^-/cm^2 as well as at the total fluence level.

Proton irradiations were carried out at the NASA Lewis Research Center cyclotron, which is operated by the Cleveland Clinic Foundation. Samples were mounted on a test fixture (fig. 3) which allowed them to be irradiated, and then submerged immediately into a dewar of liquid nitrogen. Desiccant was placed at the bottom of the dewar so that when the nitrogen diffused out of it, the samples remained dry. J_c was measured on the samples without disturbing them on the test fixture.

Both the electron and proton fluences tested here are equivalent to what the material might see in the hottest portions of the Van Allen belts during a period of maximum solar activity for about a year. Typical radiation levels in low earth orbit may be as many as seven orders of magnitude lower.

RESULTS AND DISCUSSION

Ambient

It has been apparent from the earliest work that these materials are sensitive to the ambient environment. Kitazawa et al. have discussed the problem in some detail (ref. 2). They find there is an enhancement of the degradation when both water and carbon dioxide are in the environment. They did not, however, quantify this deterioration for samples which were simply exposed to the ambient laboratory air. It was important for us to determine the effect of the ambient air on our samples both as an internal control measure, and also as a guide for other researchers.

As expected, degradation of the normal state resistivity when the samples were stored at room temperature was observed. As is illustrated in Fig. 4, the resistivity increases by about 4 percent over a seven week period. The amount of degradation is no doubt dependent on the surface to volume ratio.

In these samples the surface to volume ratio was about 40 cm^2/cm^3. For films and fibers this number would be much higher, and degradation would be expected to be faster.

We also monitored the resistivity of samples stored in a desiccator with a $CaSO_4$ drying agent. Although stability was improved, Fig. 4 shows that there was still a normal state resistivity increase of nearly 2 percent over this same time period.

Vacuum

Although it has been apparent that the oxygen in $Ba_2YCu_3O_{7-x}$ is labile at high temperatures, little is known about the behavior of the material in vacuum for long time periods at room temperature and below. Kroeger et al., however, have observed compositional and morphological changes on fracture surfaces with time in vacuum at room temperature using Auger electron spectroscopy even in the absence of electron beam excitation (ref. 3).

The tests in this study show very little, if any, degradation in the normal state resistivity of the samples on exposure to vacuum (fig. 4). The changes reported by Kroeger et al. occur very near the surface and have negligible effect on the bulk, at least over time spans of a few weeks. However, the vacuum under which our tests were conducted was not as great as that in the other study. It is still possible that, in deep space vacuum after several years, effects could surface. These would most seriously effect thin films, with their high surface to volume ratios.

Humidity

There have been many reports in the literature about the instability of $Ba_2YCu_3O_{7-x}$ to water. Interestingly, it has been reported to be inert to submersion in cold water for several hours (ref. 4), though even slight heating (35 °C) causes a rapid decomposition into BaY_2CuO_5, $Ba(OH)_2$, and O_2. The $Ba(OH)_2$ then reacts with CO_2 to form $BaCO_3$ (ref. 5).

A more troubling problem than immersion is the reaction with water vapor in the air. Dexin et al. reported that exposure of a sample in air at 100 percent humidity at 60 °C for 20 min resulted in needle crystals growing from crystal grain edges (ref. 6). We saw the same reaction. They further report that T_c (zero) drops with time (to 50 K after 80 min), but that T_c (onset) is nearly unaffected.

This study was similar to that of Dexin et al., except that the temperature of the reaction was also varied. The results are summarized in figure 5, which shows the log of the resistivity/initial resistivity (assuming no dimensional changes) as a function of time at 30, 40, and 50 °C, 100 percent humidity.

If normal state conductivity is used as an indicator of the extent of reaction, interesting trends emerge. When raising the temperature in these systems there are two effects; raising the average kinetic energy of the molecules, and increasing the vapor pressure of the water. A change from 30 to 50 °C changes the kinetic energy of the gas phase molecules by less than

7 percent, but increases the vapor pressure by a factor of three. If, therefore, increasing the temperature is taken to have only the effect of changing the available concentration of water molecules, the data are found to lie along a straight line on a graph of vapor pressure of water as a function of the reciprocal of the time to reduce the conductivity by half (fig. 6). This implies a second order reaction.

This surprising result can lead to many different mechanisms. We speculate that perhaps the species which initiates the decomposition is not H_2O, but actually the H_3O^+ -- OH couple, with the OH^- abstracting the Ba, and the H_3O^+ acting to stabilize the activated transition complex. The fact that the compounds show little decomposition when exposed to NaOH solutions suggests that the H_3O^+ is important (ref. 7).

Perhaps a second order mechanism simply reflects the divalency of the barium ion, which must capture two water hydroxide ions to be debonded from the $Ba_2YCu_3O_{7-x}$ lattice. Without more data, there are few constraints on other scenarios.

Electron Irradiation

There have been at least two studies of the effects of electron irradiation on $Ba_2YCu_3O_{7-x}$. Maisch et al. irradiated thick films with very high energy electrons (55.7 MeV), and found at high fluences (100 mrad) that the room temperature resistivity increased by a factor of 2 to 3, and became more semiconductor-like in its temperature dependence. They observed no shift in T_c (onset), but a decrease of about 5 K in T_c (zero)(ref. 8).

Recently, Adem et al. reported a study using 1 MeV electrons at fluences of 6.5×10^{11} and 1.7×10^{15} e^-/cm^2 (ref. 9). They also reported a decrease of T_c (zero) to 52 and 40 K respectively, no substantial change in T_c (onset), and that the samples behaved more semi-conductor-like. They further reported increasing disorder in the lattice and rounding of grain boundaries caused by the irradiation.

In this study we looked at J_c as a function of irradiation with 1 MeV electrons (fig. 7). It was found that fluences up to 9.7×10^{14} e^-/cm^2 degraded J_c (77 K) by no more than about 2 percent over the control samples. A limited number of samples (six) were used, so even this degradation could be within the deviation of the samples. The control samples showed some degradation also, probably due to exposure to the air during the irradiations.

Proton Irradiation

The effects of proton irradiation at a variety of proton energies has been studied in a preliminary way. Willis et al. studied sintered pellets of $Ba_2GdCu_3O_{7-x}$ and $Ba_2EuCu_3O_{7-x}$ irradiated with 800 MeV protons at 90 °C (ref. 10). T_c (onset) for both compounds dropped at a rate of (1.7 K)/10^{17} p^+/cm^2). SQUID measurements showed that flux expulsion decreased from 20 percent to less than 10 percent after 10^{17} p^+/cm^2. Intragrain J_c was found to increase by a factor of three in zero field.

In another study, Maisch et al., studied thick films (100 μm) of $Ba_2YCu_3O_{7-x}$ irradiated with protons of 63 MeV up to a fluence of 1.09×10^{14} p^+/cm^2, and with 6 MeV protons to a fluence of 5.0×10^{13} p^+/cm^2 (ref. 6). With the 63 MeV samples it was found that at fluences as low as 7.8×10^{12} p^+/cm^2, there was no T_c (zero) above 4.2 K. Except for a small random disordering of the lattice, there was no change on the structure as detected by x-ray diffraction. It was difficult to interpret the 6 MeV proton data because the penetration depth (150 μm) was similar to the sample thickness.

In this study, 42 MeV proton irradiation revealed a stability effect that was not observed in any of the other tests. There were two sample types for these tests. Samples produced at NASA had large grains and lower initial J_c values (≈30 A/cm^2). Samples produced at Ferro had small grains and higher initial J_c values (≈240 A/cm^2). The 42 MeV protons proved much more damaging to the large grain samples (fig. 8). After a fluence of 8.7×10^{14} p^+/cm^2 the small grain samples showed essentially no degradation, whereas the large grain samples degraded to about one-third of their original J_c. We think that the larger grain size makes the NASA produced samples much more susceptible to proton radiation than the Ferro produced samples.

CONCLUSIONS

There are some practical handling techniques suggested by this study. First, this study confirms the results of others that atmospheric water vapor should be avoided. Although the material does not degrade so rapidly as to prohibit experimentation outside of a glove box (at least at this surface to volume ratio), it does suggest that long term storage in vacuum is superior to storing them in a desiccator.

The irradiation experiments indicate that, except for the large grained samples, the materials are reasonably hard to the radiation they are likely to see in space. What their lifetime can be expected to be is difficult to determine without detailed knowledge of the specific mission. Additional studies are required to determine the failure points for both proton and electron radiation, and to verify if the grain size effect is real.

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Figure 1. - Sample holder for making four-point resistivity measurements on ceramic bars of $Ba_2YCu_3O_{7-x}$.

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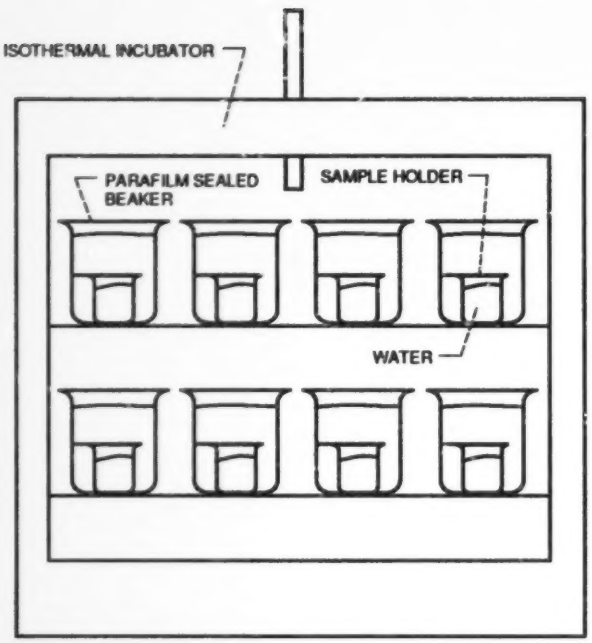

Figure 2. - Humidity chambers used for testing the stability of ceramic bars of $Ba_2YCu_3O_{7-x}$.

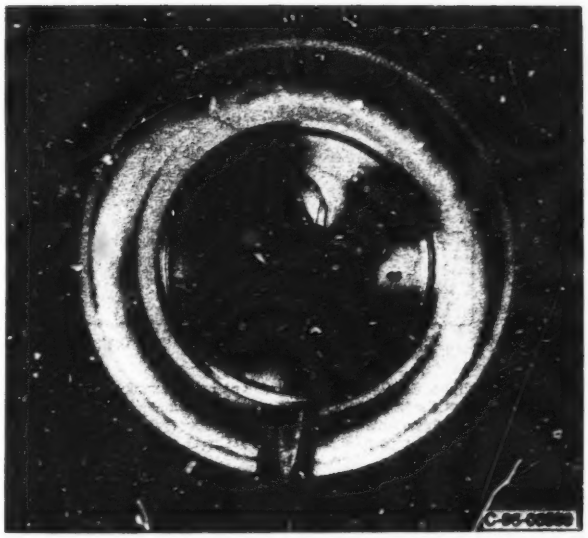

Figure 3. - Test fixture to irradiate ceramic bars $Ba_2YCu_3O_{7-x}$ in a cyclotron, and then measure their critical current density.

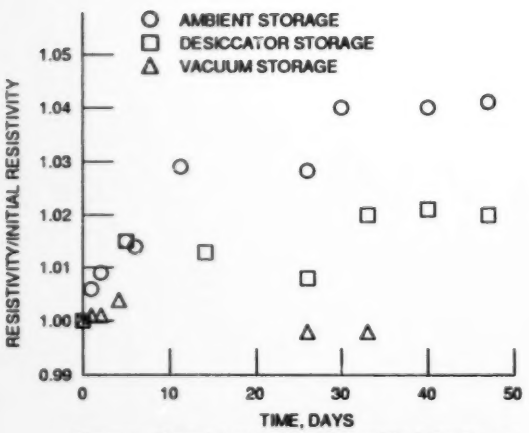

Figure 4. - Degradation of ceramic bars of $Ba_2YCu_3O_{7-x}$ on exposure to ambient conditions, desiccated air, or vacuum over time.

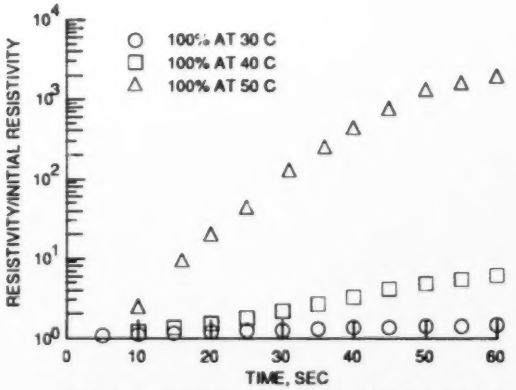

Figure 5. - Degradation of ceramic bars of $Ba_2YCu_3O_{7-x}$ on exposure to 100 percent humidified air, at 30, 40, and 50 °C over time.

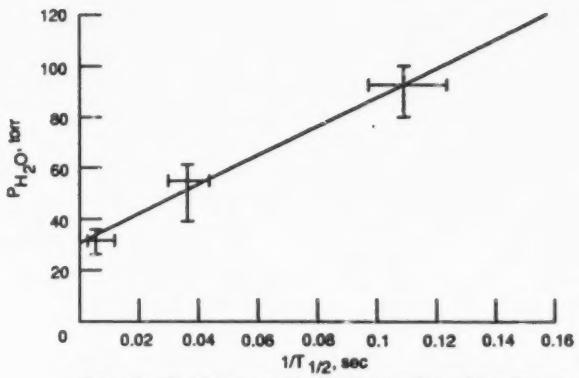

Figure 6. - Partial pressure of water as a function of the reciprocal of the time to degrade the sample conductivity by half. Linearity in this graph indicates a second order chemical reaction of water with the ceramic bars of $Ba_2YCu_3O_{7-x}$.

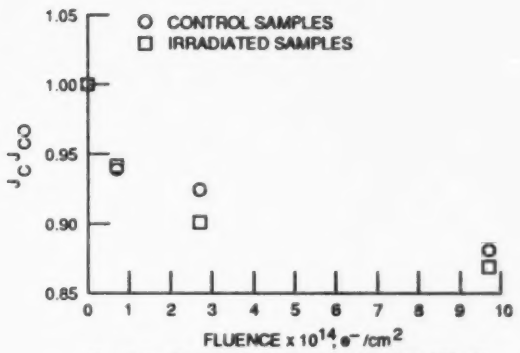

Figure 7. - Degradation of ceramic bars of $Ba_2YCu_3O_{7-x}$ on exposure to 1 MeV electron irradiation.

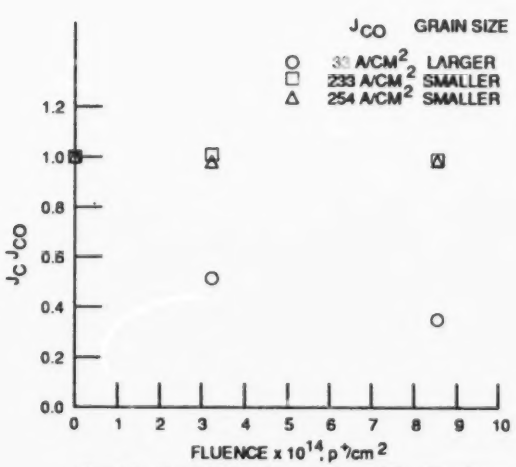

Figure 8. - Degradation of ceramic bars of $Ba_2YCu_3O_{7-x}$ on exposure to 42 MeV proton irradiation.

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16. Abstract

Small bars (1 by 1 by 14 mm) of ceramic $Ba_2YCu_3O_{7-x}$ were fabricated and subjected to environments similar to those that might be encountered during some NASA missions. These conditions include ambient conditions, high humidity (100 percent humidity up to 50 °C), vacuum (10^{-4} Pa), and high fluences of electrons (9.7×10^{14} e^-/cm^2) and protons (8.7×10^{14} p^+/cm^2). The normal state resistivity or critical current density (J_c) were monitored during these tests to assess the stability of the material under these conditions. When normal state resistivity is used as a criterion, the ambient stability of these samples was relatively good, exhibiting only a 2 percent degradation over a 3 month period. The humidity stability was shown to be very poor, and to be a steep function of temperature. Samples stored at 50 °C for 40 min increased in normal state resistivity by four orders of magnitude. Kinetic analysis indicates that the degradation reaction is second order with water vapor concentration. It is suspected that humidity degradation also accounts for the ambient instability. The samples were stable to vacuum over a period of at least 3 months. Degradation of J_c (defined as the current density that produced a potential greater than 1 μV/mm) in an 1 MeV electron fluence of 9.7×10^{14} e^-/cm^2 was determined to be no more than about 2 percent. Degradation of J_c in a 8.7×10^{14} p^+/cm^2 of 42 MeV protons was found to be grain size dependent. Samples with smaller grain size and initial J_c (77 K) of about 240 A/cm^2 showed no degradation, while that with larger grain size and an initial J_c (77 K) of about 30 A/cm^2 degraded to 37 percent of its original value.

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